SMART STRUCTURAL COMPOSITES WITH PIEZOELECTRIC MICRO-CONSTITUENTS

Final Report

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December, 1992

U.S. ARMY RESEARCH OFFICE

GRANT NUMBER: DAAL03-89-G-0086

CENTER FOR MATERIAL RESEARCH AND ANALYSIS
AND DEPARTMENT OF ENGINEERING MECHANICS
UNIVERSITY OF NEBRASKA-LINCOLN

REPORT DOCUMENTATION PAGE

Form Approved
OMB No. 0704-0188

1. AGENCY USE ONLY *(Leave blank)*

2. REPORT DATE
December 1992

3. REPORT TYPE AND DATES COVERED
FINAL REPORT SEP 89- DEC 92

4. TITLE AND SUBTITLE
SMART STRUCTURAL COMPOSITES WITH PIEZOELECTRIC MICRO-CONSTITUENTS

5. FUNDING NUMBERS
DAAL03-89-G-0086

6. AUTHOR(S)
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7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)
DEPARTMENT OF ENGINEERING MECHANICS
UNIVERSITY OF NEBRASKA-LINCOLN
LINCOLN NE 68588-0347

8. PERFORMING ORGANIZATION REPORT NUMBER

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)
U. S. Army Research Office
P. O. Box 12211
Research Triangle Park, NC 27709-2211

10. SPONSORING/MONITORING AGENCY REPORT NUMBER
ARO 27198.1-MS-SM

11. SUPPLEMENTARY NOTES
The view, opinions and/or findings contained in this report are those of the author(s) and should not be construed as an official Department of the Army position, policy, or decision, unless so designated by other documentation.

12a. DISTRIBUTION/AVAILABILITY STATEMENT
Approved for public release; distribution unlimited.

12b. DISTRIBUTION CODE

13. ABSTRACT *(Maximum 200 words)*
The research culminated into the feasibility of smart structural composite materials, in which the microstructure is modified with the introduction of a piezoelectric constituent. Greater success was achieved in the sensing aspect of smart properties, enabling the material to have an inherent property of health monitoring. By sensing and quantifying elastic strain, the material can monitor its dynamic state (vibration), degradation and damage. In actuation, the material is readily suitable for active vibration control and manipulative damping. One out-growth of the results was an active fibrous sensor, which offers a readily viable alternative to a passive fiber-optic sensor as it is marred with breakage problems. A new concept of piezo-electric emission, analogous to acoustic emission, emerged which imparts the material ability to sense stress waves. This ability can be used to locate the regions of delamination and local fractures through fabrication to operational life of structure. Techniques were developed for deposition of thin piezoelectric (ZnO) film on carbon and metal fibers. Sensing and actuation properties were imparted into carbon fiber composites. The results demonstrated self-sensing of strain and damage measurement in beam and shell elements. The concept is ready for technological advancement and application.

14. SUBJECT TERMS
SMART MATERIALS, STRUCTURAL MATERIALS
CARBON FIBER COMPOSITES

15. NUMBER OF PAGES
12

16. PRICE CODE

17. SECURITY CLASSIFICATION OF REPORT	18. SECURITY CLASSIFICATION OF THIS PAGE	19. SECURITY CLASSIFICATION OF ABSTRACT	20. LIMITATION OF ABSTRACT
UNCLASSIFIED	UNCLASSIFIED	UNCLASSIFIED	UL

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FORWARD

The financial support and technical guidance from the Material Science Division of the U.S. Army Research Office is gratefully acknowledged. In particular, thanks are due to Drs. Andrew Crowson and Edward S. Chen for their encouragement and support in this research and their leadership in the advancement of the field of Smart Materials and Structures.

The seed grant and subsequent matching financial support from the Center for Materials Research and Analysis has been paramount in the success of this research project. The inter-disciplinary research was only possible through the help of co-investigators Drs. N. J. Ianno, D. C. Timm and R. O. Dillon. Support and guidance from Professors D. J. Sellmyer, R. J. De Angelis and M. Negahban is also gratefully acknowledged. Several graduate and undergraduate students were supported under this program, and their contribution is acknowledged as well.

SMART STRUCTURAL COMPOSITES WITH PIEZOELECTRIC MICRO-CONSTITUENTS

Background

The concept of smart materials offers a fresh approach to the solution of a variety of problems concerning materials and structures. Furthermore, it affords some capabilities that would not be practical otherwise. Through the feature of health monitoring, it brings in new schemes to enhance performance and reliability. At present the field of smart materials is in its embryonic stage. Success has primarily been achieved through the embedded sensors and actuators, which have formed a transitional phase to true smart materials. The approach has been to take existing hardware and devices, then miniaturize and incorporate them into materials and structures. The current activity in smart materials usually involves a single function, typically strain sensing (fiber optics) or stiffness variation (SMA and ERF). Smart skins, layers and films of piezo-electric materials, for sensing and actuation, have been found useful in active control.

The objective of our research was to develop smart structural materials with inherent sensing/actuating properties through microstructural design beyond the technique of embedded sensors. The research strived to synthesize "sensitive" carbon filaments by coating thin films of piezoelectric zinc oxide thereby imparting intrinsic features of sensing and actuation. The demonstration of the smart capabilities was planned through testing and evaluating vibration and damage in structural elements. The research was divided into two major activities which progressed in parallel; 1. Thin film deposition for synthesizing carbon fibers, and 2. Development of smart attributes and testing.

THIN FILM DEPOSITION AND SYNTHESIS OF SENSING/ACTUATING FIBERS

Piezoelectric layers, in the form of smart skins, have been the subject of studies for sensing and actuation, particularly for active vibration control. Our approach is to synthesize sensing and actuating constituents by thin film deposition of piezoelectric Zinc Oxide. A carbon fiber is typically 10 μm in diameter, and thickness of the film is one order of magnitude lower. Thus we can say that the modification is microstructural in nature, as compared to embedded sensors/actuators. In addition, thin film deposition results in a better crystalline structure and higher purity, both increasing the coupling coefficient of piezoelectric material. Also, complex device characteristics can be realized through etching and multiple layer deposition. In some particular applications, sensors can be etched on outer or inner layers.

Piezoelectric Materials

There are a variety of piezoelectric materials in use. Crystalline Zinc Oxide is widely used for high frequency acoustic devices requiring thin piezoelectric films. The low dielectric

constant of Zinc Oxide makes it best-suited for this application of very thin film. Both the magnetron sputtering and pulse laser deposition were used to coat carbon and metallic substrates.

Deposition of Piezoelectric Thin Film

A literature search found no prior work on piezoelectric film deposition on carbon fiber. We chose to start sputtering thin piezoelectric films on small strips of brass, stainless steel and highly oriented pyrolytic graphite (HOPG). Later wires of Ni-Cr and copper were sputtered before going on to deposition on carbon fibers.

Magnetron Sputtering: A sputtering diode system with a ZnO target was used to obtain well-oriented ZnO films. A planar magnetron sputter system with a pure zinc target was also used. The following sputtering conditions culminated through a few iteration of the relevant parameters:

oxygen\argon mixture	80:20
substrate temperature	25-200 °C
rf or dc power	300 Watts
deposition pressure	7 mTorr
target-substrate spacing	4.5 cm

The weight-gain and area of the films were used with the density of bulk ZnO to calculate coating thickness. Both Zn and ZnO targets gave deposition rates of approximately 1.5 microns per hour, and typical film thicknesses were 0.75 microns. The films deposited from ZnO on brass and HOPG were characterized by x-ray diffraction. The only ZnO peak detected was 002. As this peak ranked third in intensity in a random powder pattern, this indicates that the films are preferentially oriented with their c-axes perpendicular to the substrate. A post annealing effect was also studied and found to be beneficial, it increased crystallization as well as film quality.

Pulsed Laser Deposition: Pulsed laser deposition (PLD) is a relatively new technique that has been used to deposit a wide variety of materials. A high density laser pulse is incident on the target which is rotated to reduce trenching. The laser pulse interacts thermally and/or photochemically with the target, evaporating its near surface layer. The evaporated species explode out from the target surface and are deposited on the substrate, forming a thin film. In our work with ZnO, we have explored a variety of wavelengths and substrate temperatures, and have found a strong correlation between laser wavelength and the minimum substrate temperature required to produce (002) oriented material. As the laser wavelength decreases, so does the minimum substrate temperature. The net result is that the pulsed laser deposition of (002) oriented ZnO using 248 nm radiation can be accomplished at room temperature. The Figure 1 shows an X-ray diffraction scan of a ZnO film deposited at 248 nm from a bulk ZnO target on to (100) oriented silicon at room temperature with a background pressure of 40 mTorr. The peaks correspond to (002) oriented ZnO and silicon. Dense pinhole-free films less than 5000 angstroms thick can easily be produced using this technique. Subsequently, we deposited

ZnO via PLD on to stainless steel strips and copper wire substrates, where an excellent piezoelectric response has been observed. We believe that this technique can be easily adapted to continuous deposition on fibers and other materials without the need for precise substrate heating.

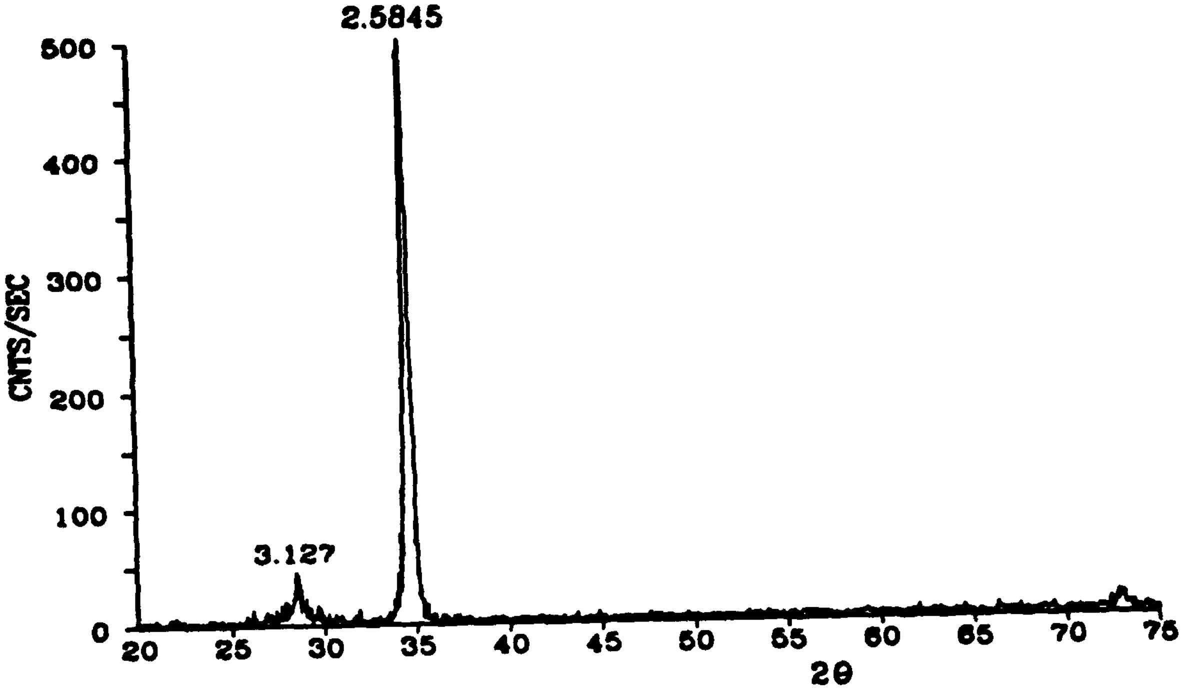

Figure 1

X-Ray Diffraction of ZnO(002) Layer on (100) Silicone Substrate

DEVELOPMENT OF SMART ATTRIBUTES AND PERFORMANCE TESTS

Sensing and actuation are two of the most important attributes a smart material should have. In structural material the primary sensing function is strain, which enables detection of deformation and damage. The actuation capability induces desired change of mechanical properties. Thus, shape changes, stiffness variation and damping can be realized on command. The specimen constituted of beam and shell elements for structural application. Before going to synthesized carbon fibers, embedded sensors were developed, affording a practical transition. This approach resulted in novel piezo-fiber sensors, that offer a viable alternative to fiber optic sensors.

Piezo-fiber Sensors

A novel sensor is developed by coating piezoelectric material on a metallic fiber. In an elegant arrangement, the metal fiber functions as one electrode, while the structural member in

which the sensor is embedded, acts as another electrode. Several of these sensors can be conveniently distributed throughout the entire structure. Similar in application to an optical fiber, the new sensor is active and does not require an external input signal. In addition, the need for subsequent conversion to an electrical output signal is eliminated. The fiber optic materials lack ductility, and thus breakage is a severe problem in their application. The sensor is well suited for fibrous composites.

Strain Sensing for Monitoring Structural Vibrations

Vibration sensing is the most typical need for structures and therefore this capability is made intrinsic in the smart material. Experimental development included both the embedded piezo-fiber sensor and synthesized carbon strands. Three types of cantilever beam specimens of exceeding sophistication were made and tested for their ability to sense vibration. Figure 2 shows the two types in which piezo-fiber sensors were embedded. Type one is a brass strip beam and type two is a Carbon-epoxy beam, both containing ZnO coated Ni-Cr wires. A brass strip layer is added in a carbon-epoxy beam to serve as a ground electrode and also to aid in handling.

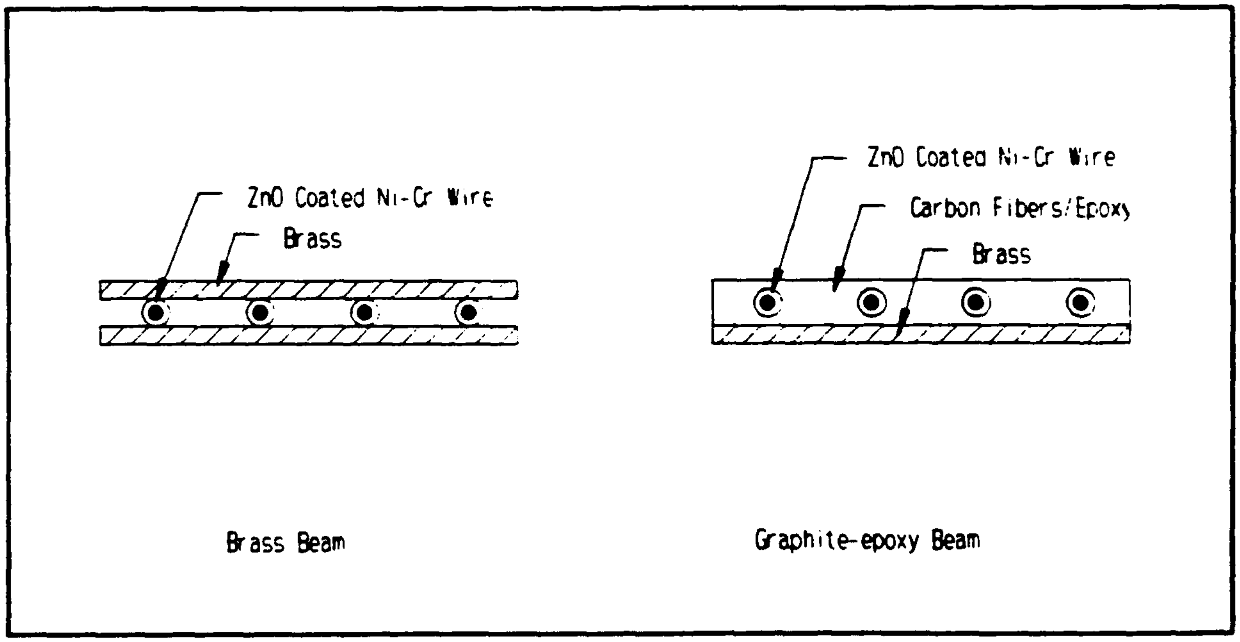

Figure 2

CROSS-SECTIONS OF BEAMS WITH EMBEDDED PIEZO-FIBER SENSORS

Each beam was tested for its inherent ability to sense vibration. The two electrodes described earlier were directly attached to a digital scope. An adequate signal was generated

without the need of an amplifier. The tests consisted of forced vibration through a shaker table as well as natural vibration by an impulse excitation. The plot of natural vibration of the cantilever beam made of brass strips is shown in Figure 3a. A damped natural vibration containing both low and high frequency modes is seen. The forced vibration test consisted of exciting the base of each beam on a shaker with a range up to 5000 cycles per second. The forced vibration response of the carbon-epoxy beam is shown in Figure 3b. The sample was excited by a shaker at various resonance modes from a frequency of 200 Hz to 5000 Hz. The top signal is from the embedded sensor, while the bottom signal is from an accelerometer mounted on the cantilever base.

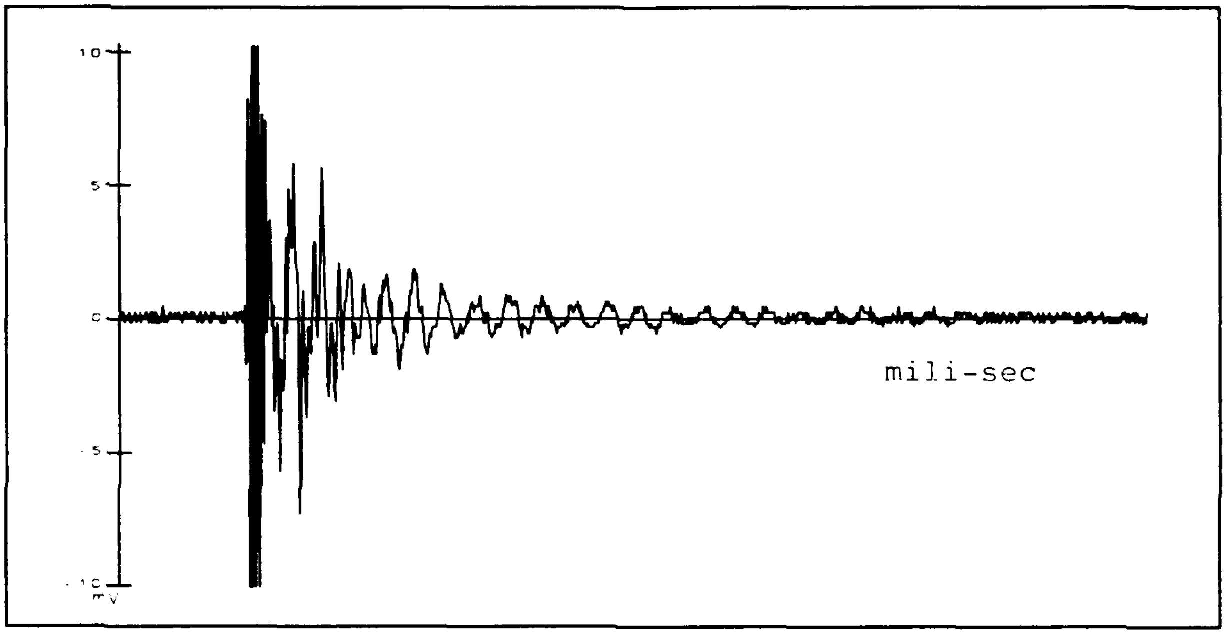

Figure 3a
Natural Impulse Response

Carbon-epoxy Composite with Synthesized Sensing Carbon Fibers

The next stage of the above work was to eliminate the embedded sensor and impart their capabilities to the carbon-fiber itself. Figure 4 shows a carbon-epoxy beam with piezo-synthesized strands laid at an angle to the longitudinal axis of the beam, simulating cross plies. The carbon of the synthesized strands acts as an electrode while the uncoated carbon-epoxy matrix acts as the ground electrode. Both electrodes were directly connected to a digital scope without any pre-amplifiers. The beam was tested for its inherent ability to sense vibration. The tests consisted of forced vibration through a shaker as well as natural vibration by impulse.

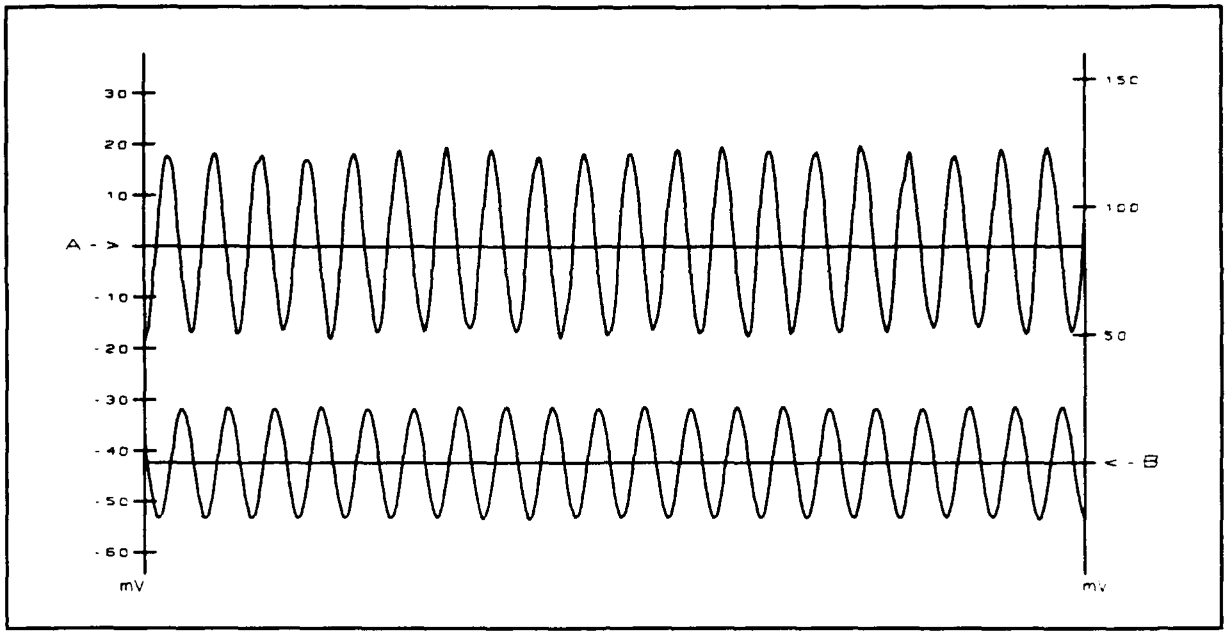

Figure 3b
Forced Response

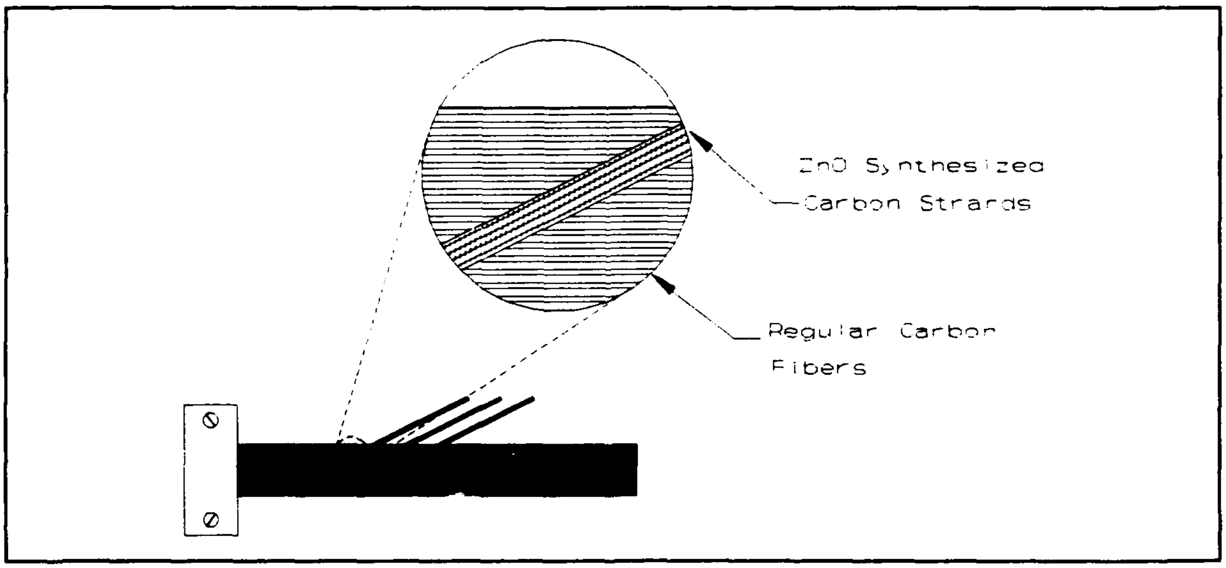

Figure 4
Carbon-Epoxy Cantilever Beam

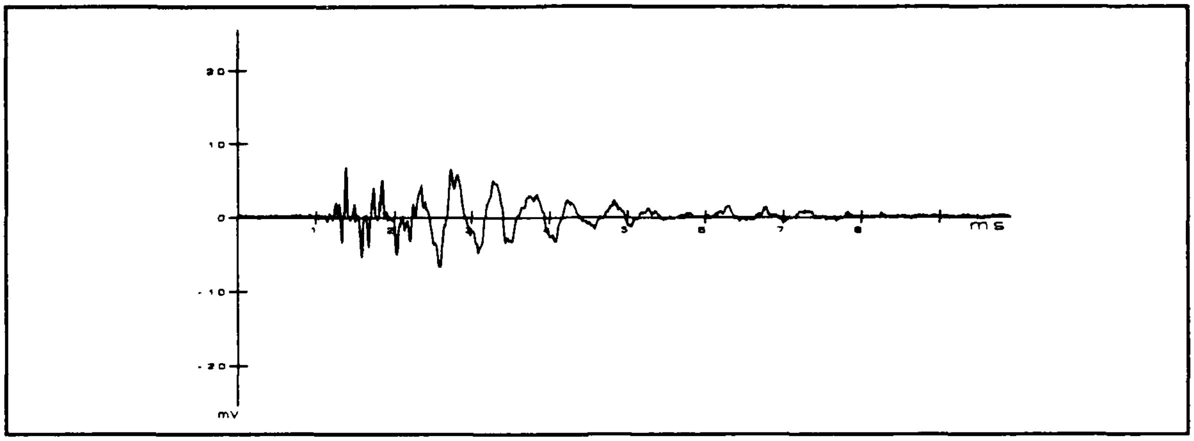

Figure 5a: Carbon Epoxy Beam; Natural Vibration

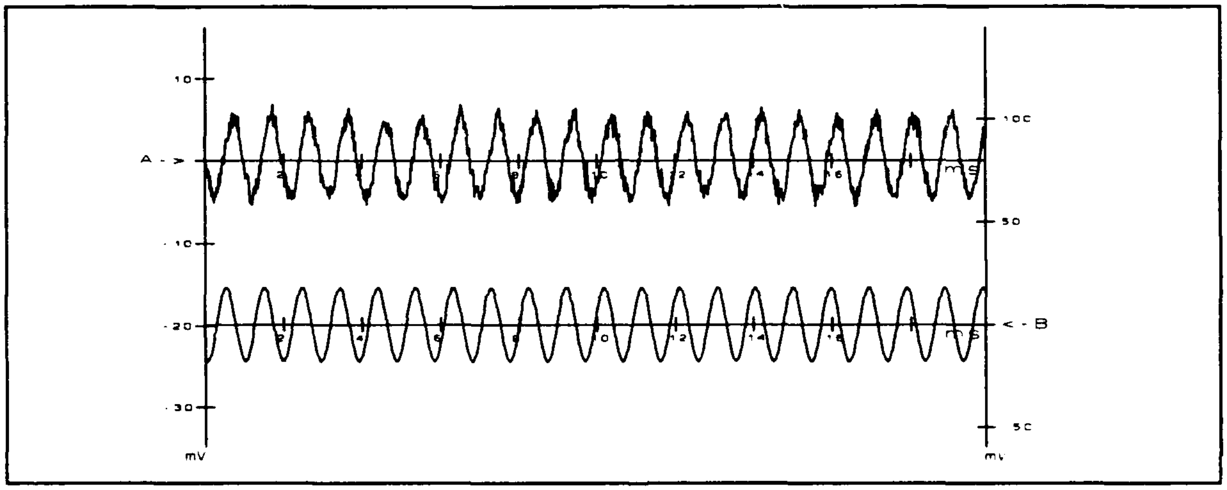

Figure 5b: Carbon Epoxy Beam; Forced Vibration

Figure 5a shows the voltage induced in the coated strand when a small ball was dropped at the end of the beam. A damped natural vibration containing both low and high frequency modes is seen. Figure 5b shows the results of a forced vibration test, where the fixed end of the cantilever beam was vibrated on a shaker. The bottom signal is from an accelerometer mounted on the fixed end of the beam, while the top signal is the voltage produced in the piezo-coated carbon strands. Thus we see the quest of an inherent smart material, realized through material synthesis rather than by way of an embedded sensor. Such a strain sensing capability is also utilized to assess the onslaught of damage, such as crack initiation, crack progression, and delaminations.

Filament Wound Composite Cylinders

Filament winding is one of the major automated manufacturing method for fibrous

composites. Having successfully tested piezo-fiber sensors in cantilever beams made of carbon composites as well as in sandwich metal beams, the sensors were next tested on filament wound composite cylinders. Figure 6 shows three Piezo-fiber sensors incorporated into a cylindrical element during filament-winding. The sample is 7 cm in diameter, 2 cm wide and 0.25 cm thick made of six plies. Both free and forced vibration tests were conducted and the vibration was measured by observing the induced voltage in the sensors. The results were similar to the beams and are not shown here for brevity. The tests for damage detection of such samples are discussed in later section.

Active Damping Control

The actuation of smart structural composites would be similar to smart skins made with piezoelectric layer. We explore a novel kind of damping which can be activated and manipulated. Unlike conventional materials, the damping can be increased or decreased under command. Different modes or frequency spectrum can be damped at the desired level, including negative damping where certain vibrations can be amplified if desired.

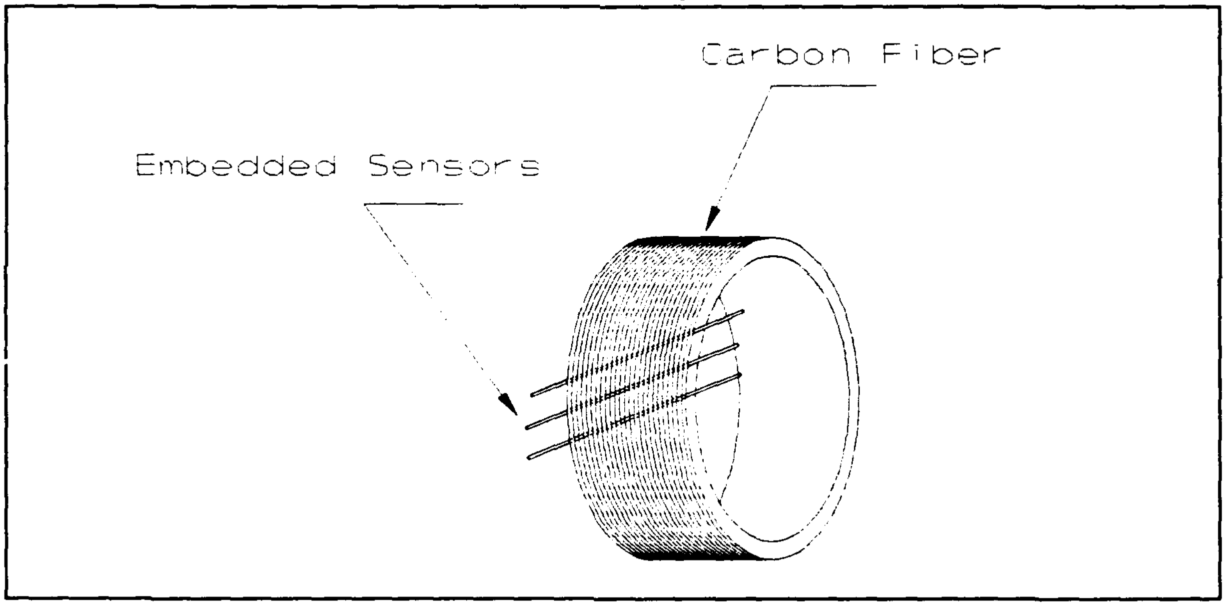

Figure 6
Filament-wound Carbon-Epoxy Cylinder

The ideas were simulated with thin sheets of piezo-ceramics, PZT-5H. Small test samples of beams, approximately 3 cm in length, were fabricated from ceramics. A rheostat was connected to the electroded ceramic beam and the resistance was varied from 2 to 10,000 Ohm. It was seen from simple test that the vibration damping of the cantilever beam can be varied by changing the energy dissipated in the rheostat. Use of charge-coupled devices, filter circuits and microprocessor can be incorporated to manipulate damping selectively. These ideas need to be further developed.

Piezo-electric Emission

In the course of research a novel property of Piezo-Electric emission (PE) was discovered. Smart materials with piezo-synthesized fibers can monitor sudden degradation and damage without any external sensors. An electric field is generated due to large local strain generated during fracture and delamination. Piezo-electric Emission is similar to acoustic emission in traditional materials. However, piezoelectric emission does not have several problems acoustic emission, such as, wave attenuation, mode conversions and appropriate location of sensors. The capability of PE adds a new feature to this class of smart material. Thus smart composites are able to monitor their own health through assessing vibration, degradation and damage.

Piezoelectric emission studies were conducted, starting with the classic acoustic emission tests of breaking Pental Pencil Lead and measuring the induced acoustic emission. Filament-wound cylindrical elements were specially fabricated for PE tests. The vibration and damage were simulated by shooting the sample with airgun pellets. In one test, the pellet ricocheted off the sample which caused severe vibration but no damage. A remarkably large signal was generated without any amplification, as shown in Figure 7. Subsequently, the pellets were shot straight on at the sample, causing delamination as well as fracture, generating the signal similar to acoustic emission signals (Figure 8). The possibility of piezoelectric emission was not envisioned at the time of proposal preparation.

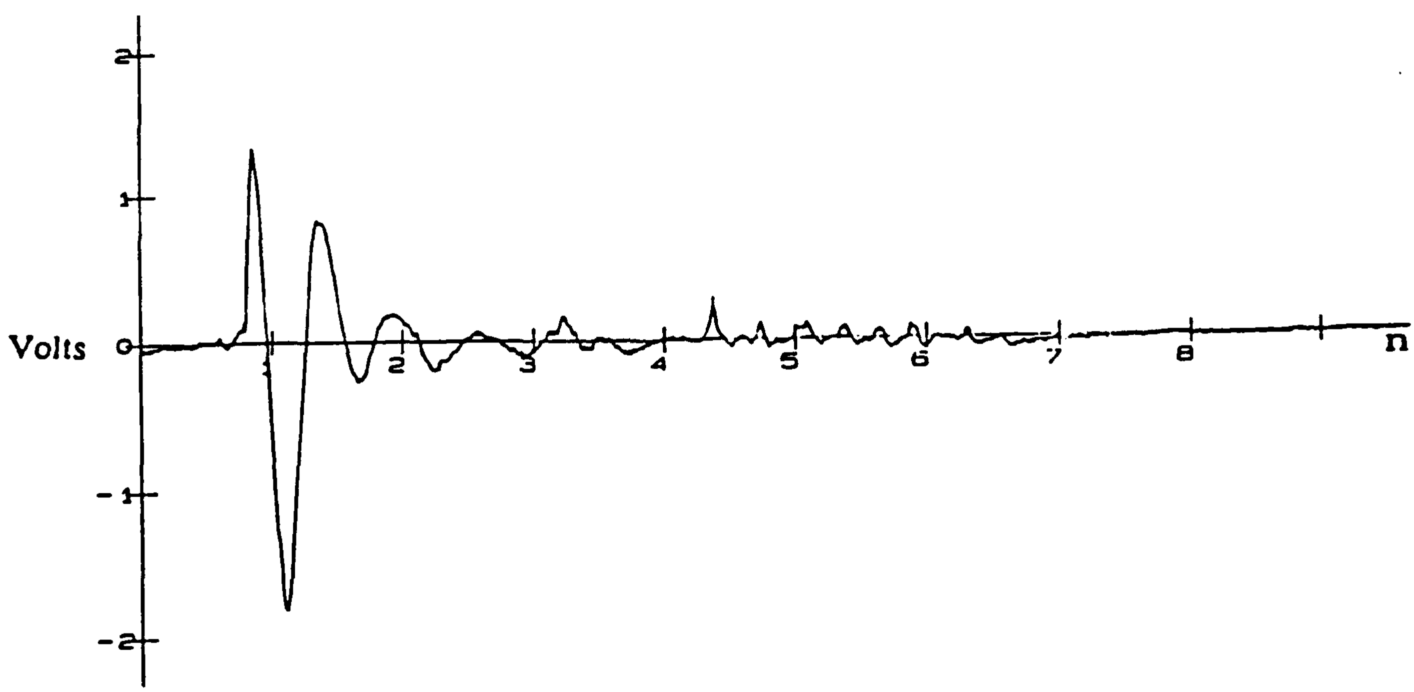

Figure 7: Pellet Impact Response

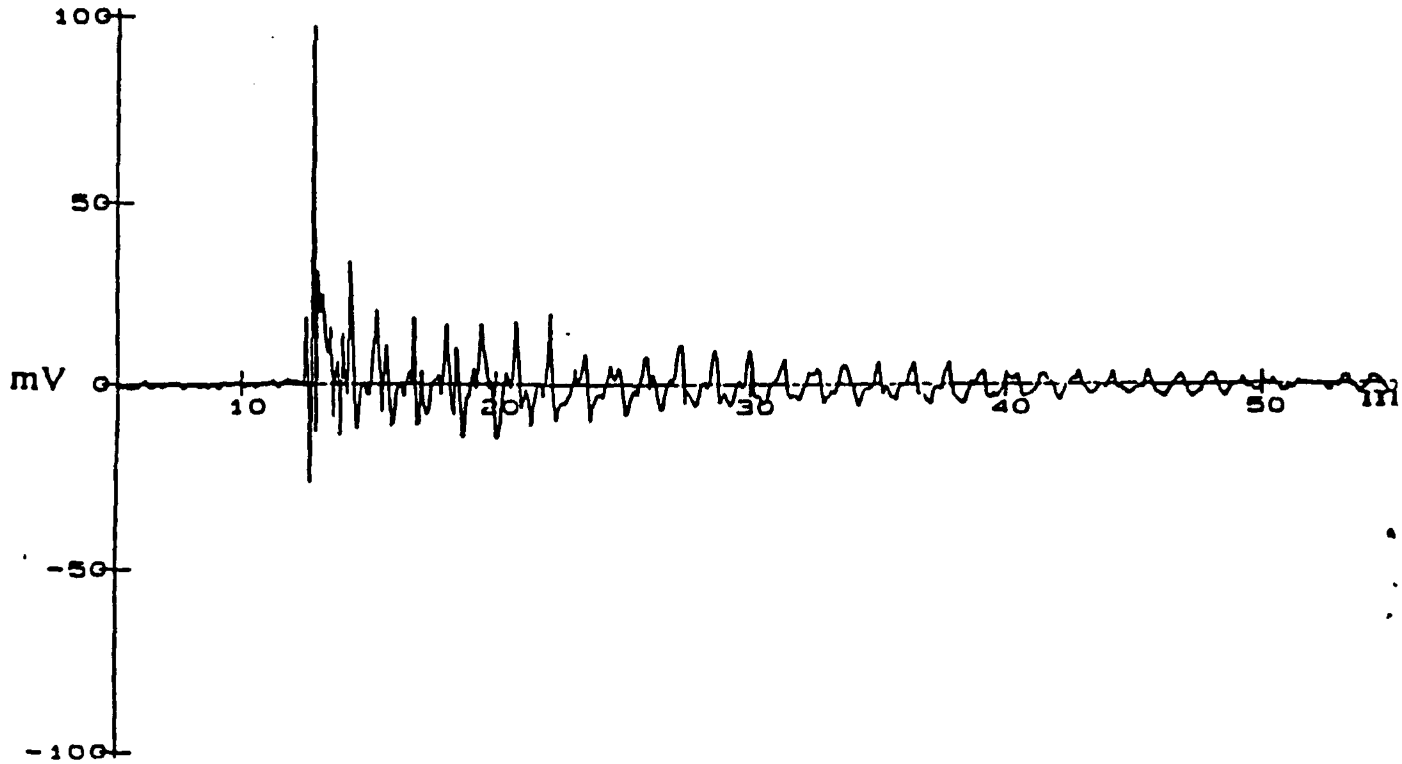

Figure 8: Fracture Induced Piezo-electric Emission

CONCLUSION

The feasibility of a true smart structural material has been demonstrated by synthesizing *carbon fibers* by piezoelectric constituents. Thus modified microstructure imparts intrinsic smart properties to the material allowing it to monitor its own health. The material can sense its dynamic state as well as fracture and delamination. Research also addressed the manufacturing aspects of the smart materials including thin film deposition and filament winding. An outgrowth of the research was a novel piezo-fiber sensor that can be embedded in materials and structures for sensing vibration and damage. This sensor offers several advantages over fiber optic sensors being tried smart structures.

REMARKS

The primary objective of the research was to strive for smart material without embedded sensors and actuators. The sensing function for health monitoring was carried out in its entirety with gratifying success. The research addressed a variety of other issues which are contained in four graduate student theses. Theoretical work involved modelling piezoelectric Zinc Oxide and analyzing the sensitized beam elements.

The accomplishment of the basic research strongly suggest that the future work should involve technological development and applications. The piezo-fiber sensor could replace the fiber optics sensors which present severe breakage problems. The synthesized carbon fibers could be used readily composite pressure vessel to monitor proof testing. The application of this research can solve various problems of nondestructive evaluation of composites parts and structures.

Technology Transfer: A seminar was presented at the Grumman Corporate Research Center and discussions were held with Dr. Gareth Knowles. Grumman has shown great interest in developing piezo-coated fiber technology as an alternative to fiber-optic sensors posing severe fabrication problems.

Report of Inventions:

1. Piezo-fiber Sensor
2. Sensitized Carbon Fibers

The patent process is started but the patents are yet to be filed.

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APPENDIX

LIST OF MANUSCRIPTS PUBLISHED UNDER ARO SPONSORSHIP

1. 'Smart Structural Composites', N. Shaikh and R. Dillon, Proceedings of US/Japan Workshop on Smart/Intelligent Materials and Systems, held March 19-23, 1990, Honolulu, Hawaii

2. 'A Novel Sensor for Adaptive and Smart Structures', N. Shaikh, Proceedings of the Conference on Active Materials and Adaptive Structures, Alexandria, Virginia, Nov. 5-7 1991

3. 'Smart Structural Composites with Inherent Sensing Properties,' N. Shaikh, Proceedings of the Conf. on Active Materials and Adaptive Structures, Alexandria, Virginia, Nov. 5-7 1991

4. 'A Novel Sensor for Adaptive and Smart Structures,'N. Shaikh, Proceedings of Japan/U.S. Conference on Adaptive Structures, Nagoya, Japan, Nov. 12-14, 1991

5. 'Characterization of Pulsed Laser Deposited Zinc Oxide,' Ianno, N.J., McConvile, L., and Shaikh, N., Smart Material Fabrication and Materials for Micro-Electro-Mechanical Systems, MRS Vol. 276, 1992

6. 'Non-resonant Systems for Adaptive and Smart Structural Control,' Shaikh, N. and Song, Y., Proceedings of 1st European Conf. on Smart Structures and Materials, Glasgow 1992

7. 'Smart Structural Composites with the Ability to Monitor Vibration and Damage,' Shaikh, N., Chen, S., Lu, Y., Timm, D., Proceedings of 1st European Conf. on Smart Structures and Materials, Glasgow, 1992

THESES

1. Smart structural Materials: Synthesis and Testing,
   N. Ranganathan, M.S. Thesis, 1991
2. Piezoelectric Thin Film Sensors for Smart Materials,
   Y. Lu, M.S. Thesis, 1992
3. Pulse Laser Deposition of Zinc Oxide,
   L. McConvile, M.S. Thesis, 1992
4. Smart Carbon-Epoxy Composite Material with Embedded Sensor,
   S. Chen, Ph.D. Thesis, 1993 (in preparation)

SCIENTIFIC PERSONNEL SUPPORTED AND DEGREES AWARDED

Dr. Nisar Shaikh, PI, Dept. of Engineering Mechanics, Dr. Natale Ianno and Dr. Rodney Dillon, Dept. of Electrical Engineering, Dr. Delmar Timm, Dept. of Chemical Engineering.

Undergraduate Students Supported: Dave Hile, Jason Hartman, David Salmen, Harold Schaeffer, Michelle Rickett, Todd Lenz, Kris Pletka
Graduate Students Supported and Degrees Granted: Hanna Lazowski, Mine Tasci, Yingdong Song, Yaojun Lu (M.S.,(EM) 1992), Xiangdong Zhang (M.S.(EM) 1991), Mehboob Kadri (M.S. (EM) 1992), S. Ranganathan (M.S.,(ME), 1992), Song Chen (Ph.D. (ChE) 1993 expected)